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Crystal structure of 4-amino-N-pyrimidin-2-ylbenzenesulfonamide

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4-Amino-N-pyrimidin-2-ylbenzenesulfonamide (sulfadiazine) was recrystallised from a mixture of ethanol and N,N-dimethylformamide. The structure of sulfadiazine was confirmed by single crystal X-ray diffraction study to be monoclinic, space group $P2_1/c$, a=13.6210(9)Å, b=5.9250(3)Å, c=14.9910(9)Å, α= γ =90°, β=114.574(2)°, T=293(2)K, Z=4, F(000)= 520, Dx=1.511 mg/m^3, absorption coefficient = 0.290mm^{-1}.

Keywords: 4-Amino-N-pyrimidin-2-ylbenzenesulfonamide, Single crystal XRD, Monoclinic structure

INTRODUCTION

Sulfadiazine is a sulfonamide antibiotic [1,2]. The sulfa drugs, derivatives of sulfonamides, are synthetic bacteriostatic antibiotics with a wide spectrum against most gram-positive and many gram-negative organisms. Sulfa drugs are antimetabolites for the bacteria which need *p*-amino benzoic acid for the synthesis of folic acid. Sulfa drugs act as competitive inhibitors of the interaction of enzyme with normal substrate p-aminobenzoic acid.In order to grow and multiply in numbers, bacterial cells need to produce genetic material. To produce DNA they require folic acid(folate). However, bacterial cells cannot take up folic acid supplied in the diet like human cells can. Instead, they synthesize it themselves.Sulfadiazine works by preventing the bacteria from producing folate. Without folate, the bacteria cannot produce DNA and so are unable to increase in numbers. Sulfadiazine therefore stops the spread of infection. The remaining bacteria are killed by the immune system or eventually die [3,4]. Sulfadiazine is sometimes given to people who have experienced a complication of a streptococcal throat infection called rheumatic fever. Sulfadiazine is used to totally eradicate streptococcal bacteria from the body and thus prevent another episode of rheumatic fever. Repeated episodes of rheumatic fever carry an increased risk of causing permanent damage to the heart [5]. Sulfadiazine is used to treat many different types of infection, such as urinary tract infections, ear infections, burns, meningitis, malaria, toxoplasmosis [6]. Sulfadiazine is not often used these days because it can cause some serious side effects such as diarrhea, upset stomach, loss of appetite, dizziness, allergic reactions like skin rash, swelling of the face, lips or tongue, joint or muscle pain [7,8].It should not be used by people who have severe liver problems, kidney problems or porphyria [9].

MATERIALS AND MEASUREMENTS

Sulfadiazine and the solvents were obtained from a commercial supplier (Sigma-Aldrich). A crystal of dimensions 0.30 × 0.20 × 0.20 mm was mounted on a glass fibre. Measurements were performed at 293 [10] on a Bruker AXS kappa apex2 CCD diffractometer using monochromated Mo-Kα radiation [11](99.6% complete). The data were corrected for absorption using the multi-scan method of 9472 intensities, 1929 were collected. [R(int)=0.0213]. The structure was refined anisotropically using SHELX-97 [12,13]. The final wR2 was 0.0719 with a conventional R1 of 0.0281 for parameters; S=1.048.

Crystallographic study of 4-amino-N-pyrimidin-2-ylbenzenesulfonamide

Single crystals suitable for X-ray diffraction studies were obtained by evaporation from an ethanol/DMF mixture, yield 97%. Crystal data**:** $C_{10}H_{10}N_4O_2S$, monoclinic [14,15], space group $P2_1/c$, a=13.6210(9)Å, b=5.9250(3)Å, c=14.9910(9)Å, α=γ=90°, β=114.574(2)°, T=293(2)K, Z=4, F(000)= 520, Dx=1.511 mg/m^3, absorption coefficient = 0.290mm^{-1}. A white crystal of dimensions 0.30 × 0.20 × 0.20 mm was mounted on a glass fibre. The selected bond lengths and bond angles are presented in Tables 2 and 3. The atoms around the sulfonamide S atom are arranged in a slightly distorted tetrahedral configuration [16]. The angle of O(2)-S(1)-O(1)= 119.37(8)°,N(2)-S(1)-C(4)=106.20(8).

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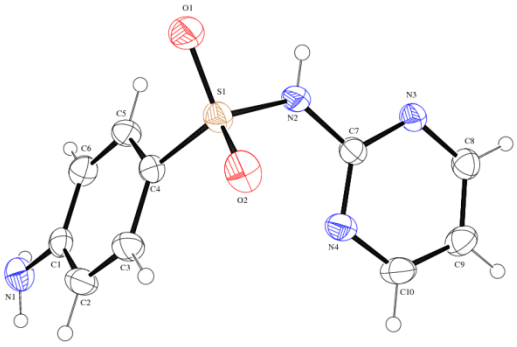

Fig. 1. ORTEP diagram of 4-amino-N-pyrimidin-2-ylbenzenesulfonamide

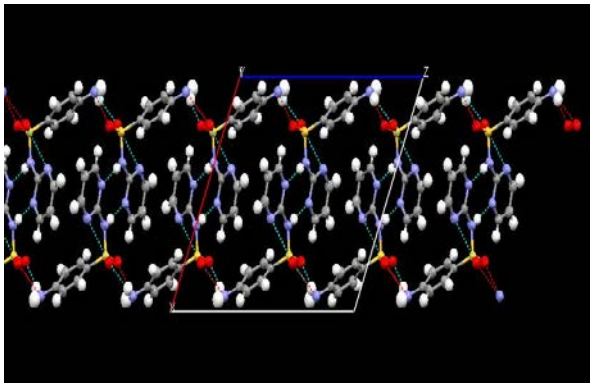

Fig.2.Packing view of 4-amino-N-pyrimidin-2-ylbenzenesulfonamide

Hydrogenbonding

The asymmetric unit contains a sulfonamide. In this crystal structure stabilized by N---H…O and N---H…N intermolecular hydrogen bonding interactions are observed. The sulfonamide moieties are centro-symmetrically paired through N---H…N hydrogen bonds involving N2---H2A…N3 base pairing leading to form a $R_2^2(8)$ ring motif. Further the base pairing units are connected through N---H…O hydrogen bonding interactions. These interactions are leading to the formation of a two-dimensional structure.

Table 1. Crystallographic data of 4-amino-N-pyrimidin-2-ylbenzenesulfonamide

Crystal data	4-amino-N-pyrimidin-2-ylbenzenesulfonamide
Crystal system, space group	Monoclinic, P21/c
Unit cell dimensions	
a,b,c	a = 13.6210(9) Å
	b = 5.9250(3) Å
	c = 14.9910(9)
α, β, γ	alpha = 90°
	beta= 114.574(2)°
	gamma = 90°
Volume	1100.26(11) $Å^3$
Calculated density	1.511 Mg/m^3
Absorption coefficient	0.290 mm^{-1}
Radition	Mo Kα
Wavelength	0.71073 Å
Temperature	293(2) K
Crystal size	0.30 x 0.20 x 0.20 mm
Theta range for data collection	2.75 to 25.00°.
Completeness to theta	25° 99.9 %
Data / restraints / parameters	1929 / 0 / 167
Limiting indices	-16<=h<=16
	-5<=k<=7
	-17<=l<=17
T_{min}, T_{max}	0.895, 0.961
Number measured reflections / unique	9472 / 1929
$\Delta\rho_{max}$/ $\Delta\rho_{min}$	0.282 and -0.290 $e.Å^{-3}$
R1/wR2	0.0281, 0.0719
R1/wR2	0.0330, 0.0765
Molecular formula	$C_{10}H_{10}N_4O_2S$
CCDC No.	917049

Table 2. Selected bond distances of 4-amino-N-pyrimidin-2-ylbenzenesulfonamide

Bond distance	(Å)	Bond distance	(Å)
C(1)-C(2)	1.391(3)	C(1)-C(6)	1.396(2)
C(2)-C(3)	1.373(3)	C(3)-C(4)	1.380(2)
C(4)-C(5)	1.385(2)	C(4)-S(1)	1.7394(16)
C(5)-C(6)	1.375(2)	C(7)-N(4)	1.325(2)
C(7)-N(3)	1.338(2)	C(7)-N(2)	1.378(2)
C(8)-N(3)	1.334(2)	C(8)-C(9)	1.373(3)
C(9)-C(10)	1.368(3)	C(10)-N(4)	1.337(2)
N(2)-S(1)	1.6471(14)	O(1)-S(1)	1.4317(13)
O(2)-S(1)	1.4232(14)	C(1)-N(1)	1.377(2)

Table 3. Selected bond angles of 4-amino-N-pyrimidin-2-ylbenzenesulfonamide

Bond angle	(º)	Bond angle	(º)
N(1)-C(1)-C(6)	120.72(18)	N(1)-C(1)-C(2)	121.12(17)
C(3)-C(2)-C(1)	121.28(16)	C(2)-C(1)-C(6)	118.11(16)
C(3)-C(4)-C(5)	120.40(16)	C(2)-C(3)-C(4)	119.62(17)
C(5)-C(4)-S(1)	119.97(12)	C(3)-C(4)-S(1)	119.62(13)
C(5)-C(6)-C(1)	121.02(17)	C(6)-C(5)-C(4)	119.55(16)
N(4)-C(7)-N(2)	118.56(15)	N(4)-C(7)-N(3)	127.05(15)
N(3)-C(8)-C(9)	122.39(16)	N(3)-C(7)-N(2)	114.39(14)
N(4)-C(10)-C(9)	123.43(17)	C(10)-C(9)-C(8)	116.48(17)
C(8)-N(3)-C(7)	115.69(14)	C(7)-N(2)-S(1)	126.59(12)
O(2)-S(1)-O(1)	119.37(8)	C(7)-N(4)-C(10)	114.93(15)
O(1)-S(1)-N(2)	101.93(8)	O(2)-S(1)-N(2)	109.70(8)
O(1)-S(1)-C(4)	109.82(8)	O(2)-S(1)-C(4)	108.92(8)
N(2)-S(1)-C(4)	106.20(8)		

Table 4. Hydrogen bond geometry (Å, º) of 4-amino-N-pyrimidin-2-ylbenzenesulfonamide

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1A)...O(1)^{i}	0.81(2)	2.19(2)	2.991(2)	170(2)
N(1)-H(1B)...O(2)^{ii}	0.88(3)	2.52(3)	3.003(2)	115(2)
N(2)-H(2A)...N(3)^{iii}	0.801(19)	2.14(2)	2.938(2)	177.5(18)

Equivalent points: (i) = x,-y-1/2,z+1/2; (ii) = x,-y+1/2,z+1/2; (iii) = -x+1,-y,-z+1

CONCLUSION

The molecular formula assigned to 4-amino-N-pyrimidin-2-ylbenzenesulfonamideby single crystal X-ray diffraction study is $C_{10}H_{10}N_4O_2S$, monoclinic, space group P21/c, a=13.6210(9)Å, b=5.9250(3)Å, c=14.9910(9)Å, α=γ=90° and β=114.574(2)°. The atoms around the sulfonamide S atom are arranged in a slightly distorted tetrahedral configuration. The angle O(2)-S(1)-O(1)=119.37(8)°, N(2)-S(1)-C(4)=106.20(8)°.

SUPPLEMENTARY DATA

Crystallographic data for the structure reported in this article are deposited at the Cambridge Crystallographic Data Centre CCDC, reference number CCDC 917049. Copies of the data can be obtained free of charge on application to CCDC 12 Union Road, Cambridge.

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КРИСТАЛНА СТРУКТУРА НА 4-АМИНО-N-ПИРИМИДИН-2-ИЛБЕНЗЕНСУЛФОНАМИД

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(Резюме)

4-Амино-N-пиримидин-2-илбинзинсулфонамид (сулфадиазин) е прекристализиран из смес на етанол и N,N-диметилформамид. Чрез рентгенов дифракционен анализ на монокристали е потвърдено, че структурата на сулфадиазина е моноклинна, пространствена група $P2_1/c$, a=13.6210(9)Å, b=5.9250(3)Å, c=14.9910(9)Å, α= γ =90°, β=114.574(2)°, T=293(2)K, Z=4, F(000)= 520, Dx=1.511 mg/m^3, абсорбционен коефициент = 0.290 mm^{-1}.